Imaging of thermal conductivity with lateral resolution of sub-micrometer using scanning thermal microscopy[1]

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[1] Paper presented at the Fourteenth Symposium on Thermophysical Properties, June 25-30, 2000, Boulder, Colorado, U.S.A.

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ABSTRACT

The measurement of thermal conductivity makes an important role in science and engineering. With the prompt development of high technologies, many objects have been in the sub-micrometer and nanometer regime in scientific research and engineering, such as microelectronics, micro-electro-mechanical systems (MEMS), new materials and biomedicine, etc. The knowledge about the thermal conductivity measurements with lateral resolution of sub-micrometer is indispensable.

In this paper the samples composed of various materials in a characteristic size of micrometer and sub-micrometer have been tested using a scanning thermal microscope (SThM). The thermal images show the apparent contrast that indicates the difference of the local thermal conductivity in the sample.

Approximate thermal resistance circuits and simplified formulas for determining the thermal tip temperature have been presented. It can be used to interpret the mechanism of heat transfer between the thermal tip and the sample and the test principle of the local thermal conductivity preliminarily.

KEY WORDS: scanning thermal microscopy; temperature; thermal conductivity; thermal resistance circuit.

1. INTRODUCTION

The measurement of thermophysical parameters, such as temperature, heat flux, heat flow rate, thermal conductivity, etc., makes an important role in science and engineering. So far, many kinds of measurement techniques have been developed to satisfy different needs in industry, science and technology. However, spatial resolutions of all those techniques are above micrometer. With the prompt development of high technologies, many objects have been in the sub-micrometer and nanometer regime in scientific research and engineering, such as microelectronics, micro-electro- mechanical system (MEMS), new materials and biomedicine, etc. Thus, the measurement technique of thermophysical parameters in micrometer and nanometer resolution is eagerly required. Therefore, the scanning thermal microscopy (SThM) was proposed accordingly [1-2]. Now, although there are many research papers and a few of products concerned SThM, the measurement mechanism is still not clearly understood, so it is necessary to make further efforts. SThM can be used in two operative modes of SThM, the temperature contrast mode and the thermal conductivity contrast mode. In this paper silicon—silicon dioxide sample, aluminum nitride sample with sintering aids are tested using a commercially available instrument (Dimension3100SPM, Digital Instrument Co.) with the function of thermal probing. The thermal image couple with the topographical image someway. The obtained thermal images show the thermal conductivity contrast with the lateral resolution of sub-micrometer. The measurement mechanism has been analyzed preliminarily using a thermal resistance circuit.

2. PRINCIPLE OF MEASUREMENT

The scanning thermal microscope is based on the atomic force microscope (AFM), the probe of AFM is mended to be a thermal probe that can perform thermal probing and some additional circuits are added into this system to make the system run in control automatically. A Si_3N_4 cantilever was used for conventional AFM experiments. Figure 1 shows the SEM image of the SThM probe tip, which has a pyramid shape tip where the thermistor is deposited[3]. When the tip is brought into contact with the surface, the resistance of the thermistor will change as a function of temperature.

The probe forms one of the legs of a Wheatstone bridge. Normally, a small current passes through the probe to detect its resistance change and its self-heating effect can be neglected. The output signal is linear with the tip temperature that relates to the temperature of the measured ample and thermal resistance between the tip and the sample surface measured[4]. As the tip contacts the sample surface, heat flows between the tip and the sample. When the sample is at room temperature while the tip temperature is higher due to laser beam irradiating on it, heat flows from the tip to the sample or vice versa when the sample is at a high temperature. So such instrument operates in two modes. One is the temperature contrast mode, where heat flows from the sample to the tip, other is the thermal conductivity contrast mode, where heat flows in the opposite direction.

2.1 Temperature contrast mode

In the temperature contrast mode, the measured sample exists in a temperature distribution because of some kind of heating, which is at higher temperature than that of thermal tip. When the probe is scanned across the sample, the heat will flow from the sample to the thermal tip. A thermal resistance circuit[5] in this mode is illustrated as Fig.2. R_c is the thermal resistance between thermistor and the base of cantilever, mainly determined by the material of cantilever and considered constant in experiments. R_t is the thermal resistance between the tip and the sample. It consists of following components: R_g, the thermal resistance due to gas conduction, R_w, the thermal resistance through water film between the tip and the sample surface, R_{ss}, the thermal resistance through solid-solid contact, R_{con}, the thermal resistance of convection and R_r, the thermal resistance of radiation. T_b is the temperature of the base of the probe, approximately equal to the circumstance temperature T_0, T_t is the temperature of tip, and T_s is that of the sample surface. The sample is considered as a heat source and the tip influences the thermal field of the sample surface very slightly during scanning.

The radius of curvature of the tip is about 100nm, and to simplify the computation, the tip contact area can be estimated in a scale of 50nm. By estimation[4-6], the thermal resistance due to gas conduction R_g is about 10^9 K·W^{-1}, the resistance through water film is estimated about 10^5 K·W^{-1}, the thermal resistance between solid and solid R_{ss} more than 10^7 K·W^{-1} and R_{con} is very large because the size of tip is so small that the convection between the tip and the sample is very hard to exist. The thermal resistance due to radiation R_r is estimated as 10^{10}K·W^{-1} when T_t=20•,T_s=60• and it is assumed that the tip and the sample are all black bodies, the angle factor equals to 1.

It is concluded that R_g, R_{con} and R_r are large enough to be ignored due to the principle of superposition of parallel resistance from the above analysis. Hence, only the thermal resistance through water film and that due to solid-solid contact is necessary to be considered, R_t is given as,

$$R_t = \frac{1}{\frac{1}{R_w} + \frac{1}{R_{ss}}} \tag{1}$$

Finally the relation between the tip's temperature and that of the sample surface can be expressed as

$$T_t = \frac{R_c}{R_c + R_t} T_s + \frac{R_t}{R_c + R_t} T_b \tag{2}$$

The output V is given as,

$$V = k \cdot (T_t - T_b) = k(T_t - T_o) = k \cdot \frac{R_c}{R_c + R_t}(T_s - T_0) \tag{3}$$

Where k is a constant determined by the gain of the apparatus, voltage of the electric bridge, the thermistor's electric resistance at room temperature and the temperature coefficient of resistance.

2.2 Thermal conductivity contrast mode

In the thermal conductivity contrast mode, the measured sample is not heated and the thermal tip radiated by the laser beam (e.g. the laser irradiation of the probe) exists in a higher temperature than that of the sample and a heat flows from tip to sample. Thermal tip acts as a thermal detector and a heat source due to the laser beam irradiating on it. The laser beam with power of 10mW illuminates on the cantilever, the tip's temperature rises up about 40• which was provided by the instrument manufacturer. The thermal resistance network model is illustrated as Fig. 3.

In Fig.3, T_b(~T_0+40^{o}C) is the temperature of the illuminated area on the cantilever, R_{c1} is the resistance between the laser spot and the tip and R_λ is the internal resistance of sample depended on its thermal conductivity. The sample temperature of the area far away from the tip can be considered to be uniform and equal to the circumstance temperature T_0. The tip's temperature can be expressed as eq. (4) according to the resistance network model,

$$T_t = \frac{R_{c1}}{R_{c1} + R_t + R_\lambda} T_0 + \frac{R_\lambda + R_t}{R_{c1} + R_t + R_\lambda} T_b \tag{4}$$

then,

$$\frac{dT_t}{dR_\lambda} = \frac{R_{c1}(T_b - T_0)}{(R_{c1} + R_t + R_\lambda)^2} \tag{5}$$

It is obvious that internal resistance of sample depended on the thermal conductivity will influence the tip's temperature and the higher the thermal conductivity is, the less R_λ goes and the lower the tip's temperature is. During the experiments in the thermal conductivity mode, the relation of the output V with R_t, R_λ is,

$$V = k \cdot (T_t - T_0) = k \cdot \frac{R_t + R_\lambda}{R_{c1} + R_t + R_\lambda}(T_b - T_0) \tag{6}$$

R_t is varied in the scanning process, and internal resistance R_λ is also varied with the scanning position if the material is inhomogeneous. The thermal conductivity contrast of micro-scale materials can be detected by this mode.

3. TEST IN THERMAL CONDUCTIVITY MODE

3.1 Experiment

Two samples have been tested in thermal conductivity mode. Fig.4 demonstrates the topographic and thermal image of a SiO_2-Si sample. The bright line in the left part of the figure is silicon dioxide, whereas other part is silicon, it means the region of SiO_2 is about 150 nm higher than that of Si. The thermal conductivity of silicon is about one order of magnitude bigger than that of silicon dioxide. When the thermal tip scans across the silicon, more heat will flow into sample than across silicon dioxide, the temperature of tip is lower and it corresponds to the dark part of the thermal image in the right part of the figure. It should be considered that the thermal image usually couples with the topographic image due to the dependence of the thermal resistance with topographic shape. Usually, the bright region in thermal image corresponds to the bright one in topographic image even though there is no thermal conductivity difference at the sample surface. The analysis of this coupling and its uncoupling is in progress. Fig.5 depicts the topographic and thermal image of an aluminum nitride (AlN) sample. The bright region in the left part of the figure is AlN, whereas another dark part is sintering aids, it means the height of AlN is bigger than that of sintering aids. The thermal conductivity of AlN is much higher than that of sintering aids. When the thermal tip scans across the AlN part, more heat will flow into sample than across sintering aids, the temperature of tip is lower and it corresponds to the dark part of the thermal image in the right part of the figure.

3.2 Discussion about experimental results

3.2.1 Coupling between the topographical image and thermal image

The coupling between the thermal image and the topographical image usually exists. It means that the higher region in the topographical image shows higher temperature in the thermal image even though the temperature is uniform over the sample. It seems that the thermal resistance between the thermal tip and the sample surface depends on the topographic form, the higher region looks like with higher thermal resistance, and then the temperature looks higher. For the sample made of different materials shown as Fig. 4 and Fig.5, the coupling effect will also exist between the topographical and thermal images in the test of thermal conductivity mode. For the first sample the silicon dioxide part is 150nm higher than the silicon part and its thermal conductivity is lower so the thermal image shows us not only the thermal conductivity contrast but also the height contrast. For the second sample, an aluminum nitride sample, the sintering aids with smaller height and lower thermal conductivity shows higher temperature in the thermal image, it seems the contrast due to the difference of thermal conductivity is more significant than that due to the coupling effect. In order to do quantitative thermal conductivity measurements with SThM, the coupling effect caused by the dependence of thermal resistance with the topography should be considered. Analysis should be done for the uncoupling to get the contrast only depended on the thermal conductivity.

3.2.2 Thermal conductivity estimation from thermal image

The thermal images obtained by SThM in the thermal conductivity mode provide the output voltage map of the Wheatstone bridge circuit shown in Fig.6. The output voltage linearly relates to the excess temperature of the tip over the room temperature, ΔT, so it is the excess temperature map as well. When the tip with higher temperature caused by laser beam irradiation scans along with the sample surface, it can be considered as a heat source supplied heat flow with a circular area as Fig. 7 shown. Assuming the thickness of the sample larger than its thermal diffusion length, by the analysis developed in [7], the relation of excess temperature with the thermal conductivity is given as eq.(7)

$$\Delta T(0,0) = P_0 / 4\sqrt{\pi}\lambda a \tag{7}$$

where $\Delta T(0,0)$ is the excess temperature at the middle point of the circular heating area, P_0 is the heat flux, λ is the thermal conductivity, a is the radius of the heating area. According to eq.(7), the thermal resistance R_λ is given as follows,

$$R_\lambda = \frac{\Delta T(0,0)}{P_0} = \frac{c}{\lambda} \tag{8}$$

where, c is a constant.

By eqs.(6) and (8),

$$\frac{(T_t - T_0)_A}{(T_t - T_0)_B} = \frac{R_{tA} + c/\lambda_A}{R_{c1} + R_{tA} + c/\lambda_A}(T_{bA} - T_0) \Bigg/ \frac{R_{tB} + c/\lambda_B}{R_{c1} + R_{tB} + c/\lambda_B}(T_{bB} - T_0) \tag{9}$$

where, subscript A denotes one materials and subscript B denotes the other materials in the sample. The heat flux supplied by thermal tip because of laser beam irradiation is considered as a constant, eq.(9) reduces to eq.(10)

$$\frac{(T_t - T_0)_A}{(T_t - T_0)_B} = \frac{R_{t\,A} + c/\lambda_A}{R_{t\,B} + c/\lambda_B} \tag{10}$$

Processing the thermal images, for example, Fig.4, the average output voltages over selected areas A for SiO_2 and B for Si shown in Fig.8 are obtained, and then, the excess temperature ratio of A area to B area are determined. The excess temperature ratio of SiO_2 area(over 2450 pixels) to Si area(over 9870 pixels) is 5.6. It is noted that this ratio include the topographical coupling, so it shows the thermal conductivity contrast partly.

4.CONCLUSIONS

(1)Using Dimension 3100 type SThM instrument, two modes of operation: temperature contrast mode and thermal conductivity mode can be applied to measure micro-scale materials and devices with the spatial resolution of sub-micrometer.

(2)Thermal resistance circuits are provided for two modes to explain the heat transfer mechanism between the thermal tip and the surface of the sample.

(3)In the thermal conductivity contrast mode, thermal images of silicon wafer with silicon dioxide line and aluminum nitride sample have been obtained.

(4)Thermal images obtained in thermal conductivity mode for inhomogeneous materials at micro-scale show the thermal conductivity contrast coupling with topography. Its explanation has been suggested.

ACKNOWNLEDGMENTS

This work is supported by the National Natural Science Foundation of China (No: 59776031)

The authors are grateful to Prof. Litian Liu of Institute of Microelectronics of Tsinghua University and Prof. Yuan Ji of Beijing Polytechnic University for providing the samples.

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FIGURE CAPTIONS

Fig.1 SEM image of SThM probe tip

Fig.2 Thermal resistance network for temperature mode

Fig.3 Thermal resistance network for thermal conductivity mode

Fig.4 Topographic (left) and thermal (right) image of SiO_2-Si sample (20×20μm)

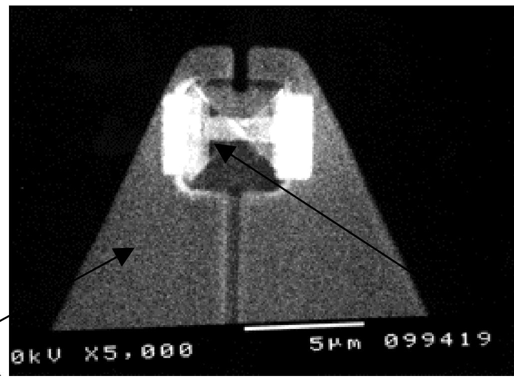
Fig.5 Topographic (left) and thermal (right) image of AlN sample (10×10μm)

Fig.6 Scheme of Wheatstone bridge circuit used in SThM

Fig.7 Scheme of sample heated by thermal tip

Fig.8 Selected areas for estimation of tip temperature

Fig.1 SEM image of SThM probe tip



Aluminum film

Thermistor

Fig.2 Thermal resistance network for temperature mode

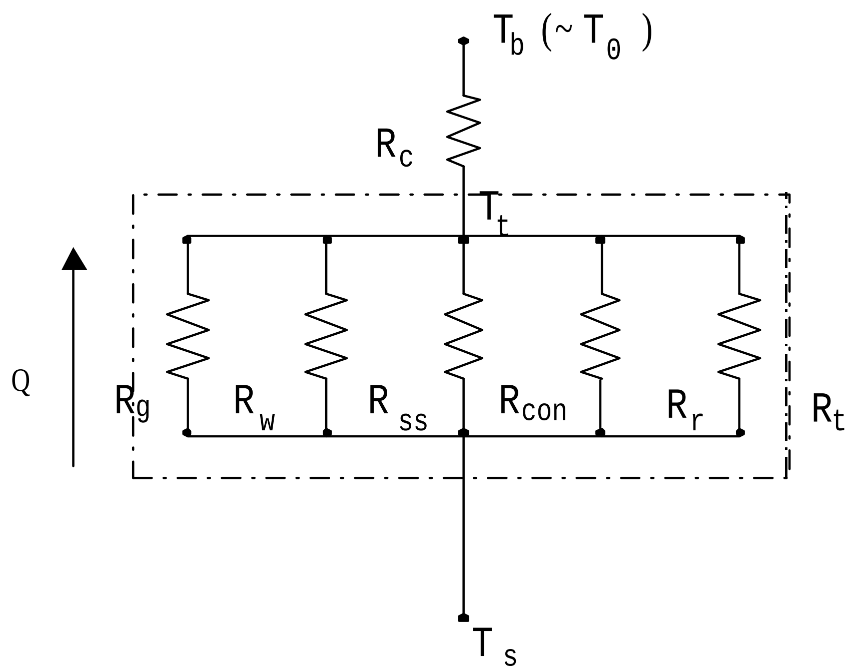


Fig.3 Thermal resistance network for thermal conductivity mode

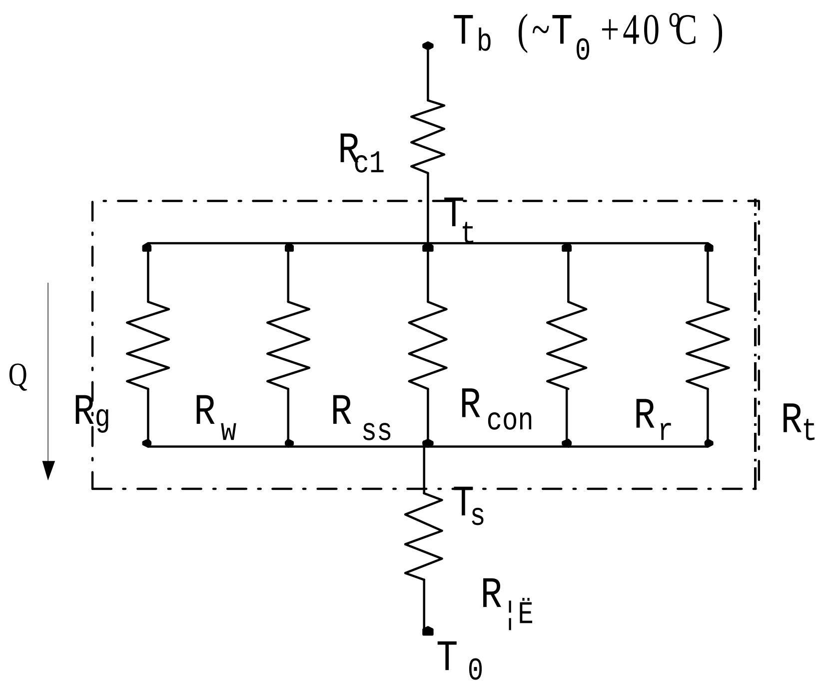


Fig.4 Topographic (left) and thermal (right) image of SiO_2-Si sample (20×20μm)

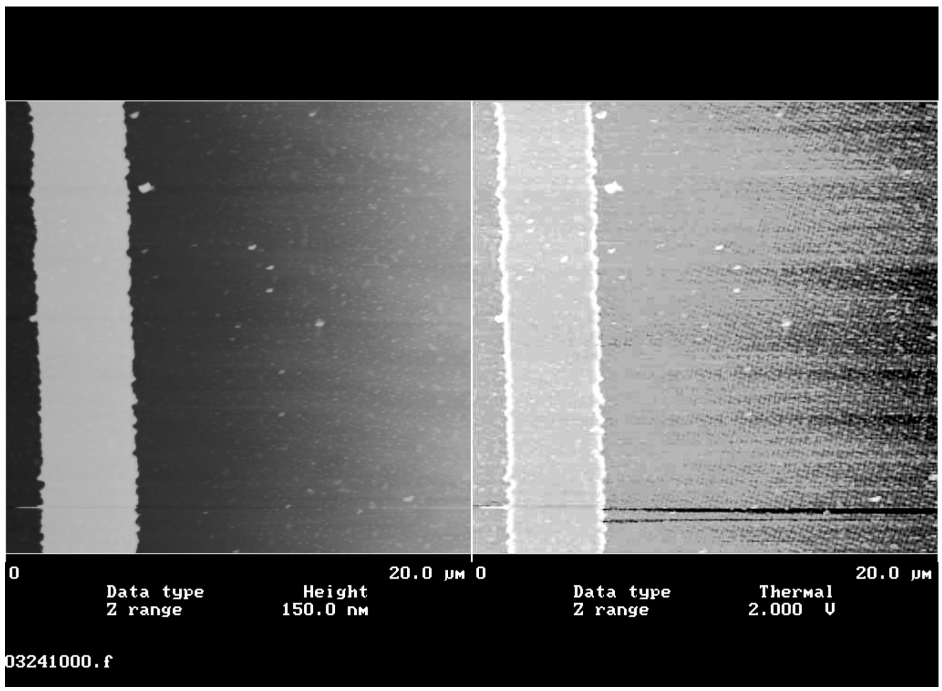


Fig.5 Topographic (left) and thermal (right) image of AlN sample ($10\times10\mu m$)

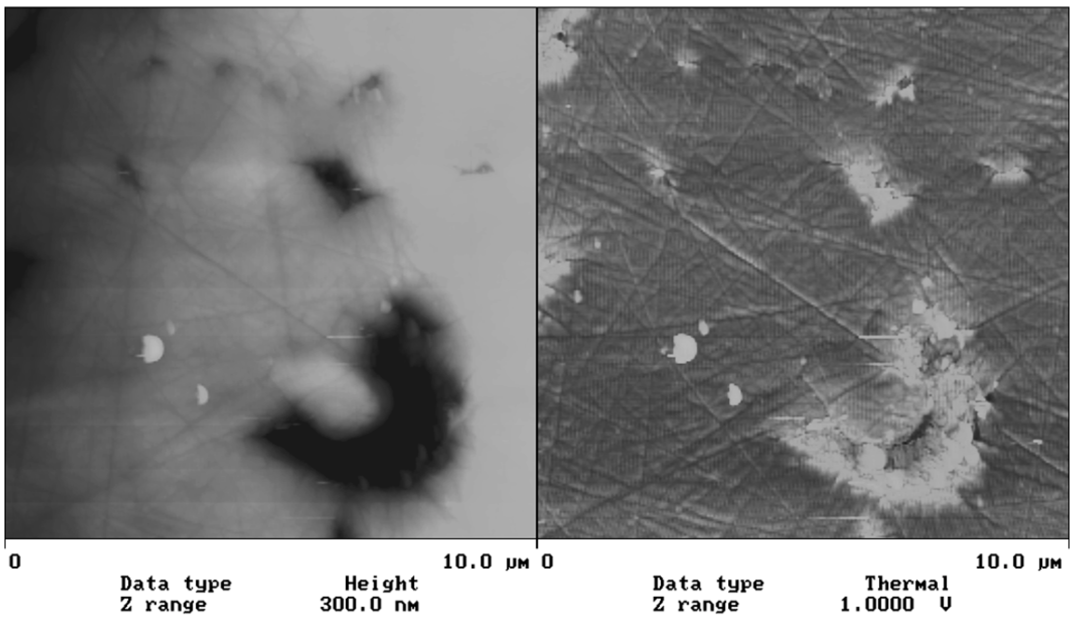


Fig.6 Schematic Wheatstone bridge circuit used in SThM

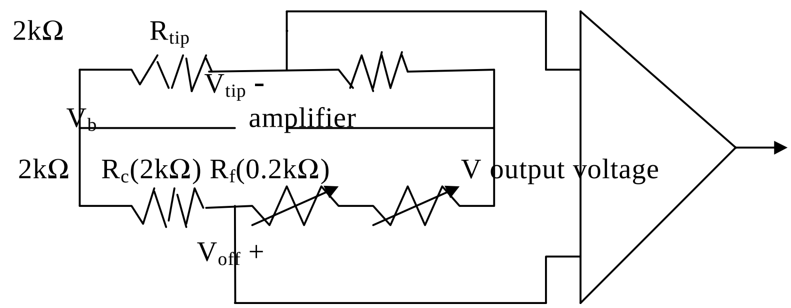


Fig.7 Scheme of sample heated by thermal tip

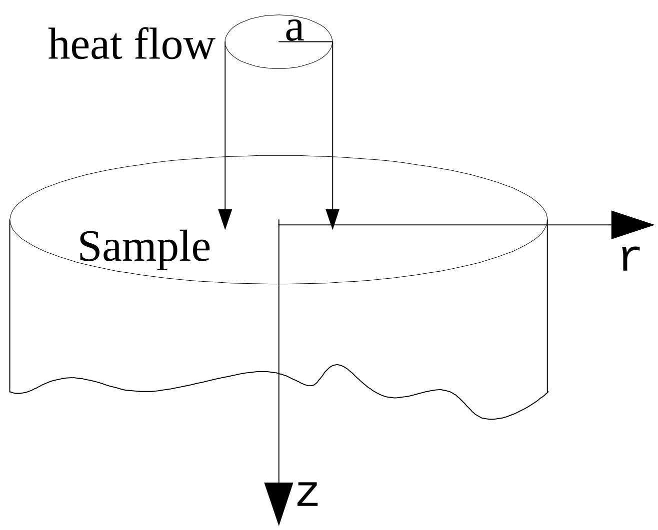


Fig.8 Selected areas for estimation of tip teperature